θ -> α Phase transition in thermally grown aluminas: mechanisms and control

Scientific Achievement

We establish that early stage development of α -Al₂O₃, a thermally grown protective oxide which first forms as a transition alumina (e.g., θ -Al₂O₃), is strongly influenced by the presence of external nucleation sites. These nucleation sites can be exploited to dramatically speed θ -> α conversion. Fine particles of α -Al₂O₃, trapped in the sample surface during the final step in the sample polishing procedure, are found to act as effective nucleation sites. These observations were obtained from in situ x-ray diffraction measurements, at APS, where the time evolution of α and θ phase oxides of Al₂O₃ was monitored as the oxides developed on β -NiAl(Zr) single crystal substrates at 1100 C in air. A simple model which incorporates known processes that occur in the phase transformation was also developed. The model provides an excellent quantitative description of the oxide evolution.

This work has been submitted to Applied Physics Letters

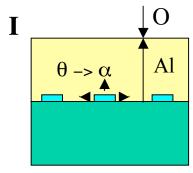
Significance

The desired oxide phase, which can provide long term corrosion protection in high temperature oxidizing environments, is α -Al₂O₃. The thickness of this oxide at completion of the θ -> α . transformation is primarily determined by the early stage θ -phase growth. The parabolic growth rate for θ -Al₂O₃ is one to two orders of magnitude faster than the rate for α -Al₂O₃. Since the probability of failure of protective oxides increases with oxide thickness, it is important to accelerate the transformation, to achieve the first transformed α -phase at a minimum thickness. Clearly, complete suppression of θ -phase growth (without introducing other deleterious effects), would be desirable for protective aluminas. Control of the transformation rate, using techniques such as reported here, can be used to extend the operating life of structural components in high temperature service.

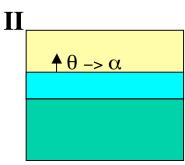
Performers

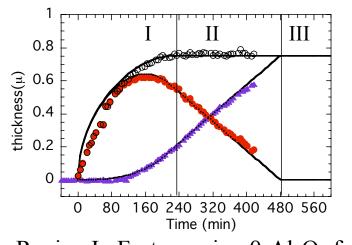
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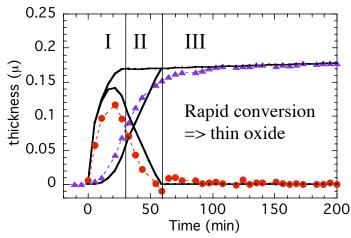
$\theta \rightarrow \alpha$ Phase transition in thermally grown aluminas

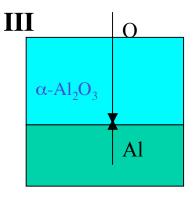


- We find: the conversion rate of θ -Al₂O₃ to α -Al₂O₃ can be controlled [Protective aluminas first form (e.g., at 1100 C) as transition aluminas]
- Rapid conversion occurs (right panel) when α -Al₂O₃ nucleation sites are added. Solid lines are model calculations.
- Longer life protective oxides can be realized with rapid phase conversion









Region I. Fast growing θ -Al₂O₃ forms first (red dots) and converts to α -Al₂O₃ at nucleation sites located at the metal/oxide interface.

Region II. The metal is covered with slow growing α -Al₂O₃ cutting off θ -phase growth. θ -> α conversion continues.

Region III. α -Al₂O₃ grows slowly at the metal/oxide interface. All θ -Al₂O₃ has been consumed.

